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# MEMORANDUM

DATE

12 January 1999

TO:

David Bennett, WAM, U.S EPA, Region X

FROM:

Michelle Turner, Chemist, WESTON, Seattle

Roger McGinnis, Semor Environmental Chemist, WESTON, Seattle

SUBJECT:

Validation of Chlorinated Pesticide Data

Laboratory Batch: K9805975

Site. Duwamish River

WORK ASSIGNMENT NO. 46-23-0JZZ

WORK ORDER NO.:

4000-019-038-5200-00

DOC CONTROL NO.: 4000-019-038-AAAK

cc:

Bruce Woods, RAP-WAM, U.S. EPA, Region X

Dena Hughes, Site Manager, WESTON, Seattle (memo only)

Kevin Mundell-Jackson, Database Management, WESTON, Seattle

The quality assurance review of three sediment samples, laboratory batch K9805975, collected from the Duwamish River has been completed. The samples were analyzed for chlorinated pesticides by Columbia Analytical Services of Kelso, Washington using EPA Method 8081. The samples were numbered:

98364003

98364011

98364012

#### **Data Qualifications**

The following comments refer to the laboratory performance in meeting the quality control criteria described in the technical specifications of the laboratory subcontract. The review follows the format described in the National Functional Guidelines for Organic Data Review (EPA OSWER Directive 9240 1-05, February 1994)

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QA Review Batch K9805975 (Chlorinated Pesticides) Site Duwamish River Page 2

#### 1 Timeliness

All samples met holding time criteria of 14 days for sample extraction and 40 additional days for extract analysis.

#### 2 GC/ECD Instrument Performance

i) Retention Time Windows

Retention times of all pesticides were within windows calculated from the initial calibration.

ii) DDT/Endrin Breakdown

The percent breakdown for 4,4'-DDT and Endrin was less than 20 percent for each compound and combined breakdown was less than 30 percent on both GC columns.

#### 3. Initial Calibration

a) Individual Standard Mixtures

Retention time windows were calculated correctly

Appropriate standards concentrations were used and peak heights of 50 to 100 percent of full scale were obtained

Calibration factor percent relative standard deviation (%RSD) met QC criteria of 20 percent for pesticides and 30 percent for surrogates

# 4. Calibration Verification

Instrument blanks and PEM samples were analyzed at the proper frequency.

The difference between actual and calculated concentrations of individual pesticides was within QC criteria of ±25 percent

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Site: Duwamish River

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# 5. Detection Limits

Instrument detection limits met project required quantitation limits with the following exceptions:

Sample	Compound	QL Goal (µg/Kg)	Reported QL (µg/Kg)
98364003	Heptachlor Epoxide	1	2
98364003	gamma-Chlordane	1	2
98364003	DDD	2	4 _
98364003	DDT	2	30
98364003	Toxaphene	10	270
98364011	Toxaphene	10	40
98364012	gamma-Chlordane	1	2
98364012	Toxaphene	10	60

Where quantitation limit goals were exceeded, undetected analytes were qualified (UI) to indicate matrix interference.

#### 6. Blanks

# a) Laboratory Method Blanks

Laboratory method blank frequency criteria were met

No target analytes were reported in laboratory method blanks

# b) Field Blanks

No field blanks were associated with this laboratory batch.

# 7 System Monitoring Compounds (Surrogates)

Surrogate compound percent recovery met quality control criteria for all samples

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# 8. Matrix Spike and Matrix Spike Duplicate

Matrix spike (MS) or matrix spike duplicate (MSD) percent recovery for the following compounds were outside QC guidelines (P-project, L-laboratory)

Sample	Compound	Percent Recovery	QC Limits
K9805842-008MS (Batch QC)	gamma-BHC (Lindane)	38	46-127 (P) 28-123 (L)
K9805842-008MS (Batch QC)	Endrin	30	42-139 (P) 39-130 (L)
K9805842-008DMS (Batch QC)	gamma-BHC (Lindane)	43	46-127 (P) 28-123 (L)
K9805842-008DMS (Batch QC)	Endrin	30	42-139 (P) 39-130 (L)

Relative percent differences (RPD) between the MS and MSD percent recoveries were within QC guidelines for all spiked compounds No action was based solely on MS/MSD data

# 9 Laboratory Control Sample (LCS)

LCS percent recoveries were outside the QC limits (P-project, L-laboratory) for the following compounds.

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Site Duwamish River

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Sample	Compound	Percent Recovery	QC Limits
K980909-LCS	gamma-BHC (Lindane)	56	70-130 (P) 40-124 (L)
K980909-LCS	Heptachlor	54	70-130 (P) 40-117 (L)
K980909-LCS	Aldrin	54	70-130 (P) 43-108 (L)
K980909-LCS	Dieldrin	57	70-130 (P) 42-127 (L)
K980909-LCS	Endrin	55	70-130 (P) 46-123 (L)
K980909-LCS	DDT	65	70-130 (P) 46-127 (L)

Results for compounds listed above were qualified as estimated (J). Undetected compounds were also qualified as estimated (UJ)

# 10. Field Duplicate Analysis

Samples 98364011 and 98364012 were field duplicates. The relative percent difference between duplicate results was within limits of 35 percent RPD for all analytes where concentrations were greater than 5 times the reporting limit

#### 11. Second Column Confirmation

The relative percent difference (RPD) in reported analyte concentration was greater than 35 percent for the primary and confirmation column for the following samples:

Sample Number	Compound	DB-5 Conc (µg/Kg)	DB-1701 Conc (µg/Kg)	RPD ,
98364003	DDE	7 57	11 22	48
98364011	DDE	1 10	1 49	35
98364012	DDT	4 07	5 60	38

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Site: Duwamish River

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Differences can arise from analytical interferences on one column. However, the relative percent differences are not deemed significant at the reported concentrations. The lower concentration was reported for each analyte.

# 12. Sample Analysis

A cursory review of raw data was performed All laboratory deliverables were present and complete. A duplicate analysis of Batch QC sample K9805842-008 was analyzed; all RPDs were less than 35 percent. The case narrative indicated that the MS recovery of Endrin for the Batch QC was outside the laboratory QC limits due to probable matrix interference. As the LCS was within laboratory QC limits, no action was taken. No other problems were noted in the case narrative

# 13. Laboratory Contact

No laboratory contact was required.

## Data Assessment

Upon consideration of the data qualifications noted above, the data are ACCEPTABLE for use except where flagged with data qualifiers that modify the usefulness of the individual values.

#### Data Qualifiers

- U The compound was analyzed for, but was not detected.
- UJ The compound was analyzed for, but was not detected. The associated quantitation limit is an estimate because quality control criteria were not met.
- J The analyte was positively identified, but the associated numerical value is an estimated quantity because quality control criteria were not met or because concentrations reported are less then CRDL or lowest calibration standard.
- R Quality control indicates that data are unusable (compound may or may not be present). Resampling and reanalysis are necessary for verification.
- N Presumptive evidence of presence of material (tentative identification).

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I - Elevated reporting limit due to matrix interference

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DCN 4000-019-038-AAAK
12 January 1999
Region X

# COLUMBIA ANALYTICAL SERVICES, INC.

# **Analytical Report**

Chent:

Roy F Weston, Inc

Project:

Duwamish River/4000-027-001-2019-38

Sample Matrix:

Sediment

Service Request: K9805975

Date Collected: 8/31/98
Date Received: 9/1/98

Units ug/Kg (ppb)

Basis Dry

# Organochlorine Pesticides

Sample Name

98364003

Lab Code.

K9805975-002

Test Notes.

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
alpha-BHC	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
beta-BHC	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
gamma-BHC (Lindane)	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND Was	
Heptachlor	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Aldrın	EPA 3550A	8081A	1	1	9/9/98	10/2/98	$ND$ $\downarrow$	,
Heptachlor Epoxide	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND IAT	₽,
gamma-Chlordane	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND 🕹	<b>J</b> g
Endosulfan I	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	·
alpha-Chlordane	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Dieldrin	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND UJ	
4,4'-DDE	EPA 3550A	8081A	1	1	9/9/98	10/2/98	8	
Endrin	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND UJ	
Endosulfan II	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
4,4'-DDD	EPA 3550A	8081A	4	1	9/9/98	10/2/98	ND UI	₿
Endrin Aldehyde	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	1
Endosulfan Sulfate	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
4,4'-DDT	EPA 3550A	8081A	30	1	9/9/98	10/2/98	ND UI	7 B
Endrin Ketone	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	,
Methoxychlor	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Toxaphene	EPA 3550A	8081A	270	1	9/9/98	10/2/98	ND UI	B

В

The MRL is elevated because of matrix interferences

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Approved By

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# COLUMBIA ANALYTICAL SERVICES, INC.

# **Analytical Report**

Client:

Roy F Weston, Inc

Project:

Duwamish River/4000-027-001-2019-38

Sample Matrix:

Sediment

Service Request: K9805975

Date Collected: 8/31/98

Date Received: 9/1/98

# Organochlorine Pesticides

Sample Name Lab Code

**Test Notes** 

98364011

K9805975-010

Units: ug/Kg (ppb)

Basis Dry

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
alpha-BHC	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
beta-BHC	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
gamma-BHC (Lindane)	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND US	
Heptachlor	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Aldrın	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Heptachlor Epoxide	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
gamma-Chlordane	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Endosulfan I	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
alpha-Chlordane	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Dieldrin	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND W	
4,4'-DDE	EPA 3550A	8081A	1	1	9/9/98	10/2/98	1	
Endrin	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND WJ	
Endosulfan II	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
4,4'-DDD	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
Endrin Aldehyde	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
Endosulfan Sulfate	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
4,4'-DDT	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND W	
Endrin Ketone	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
Methoxychlor	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Toxaphene	EPA 3550A	8081A	40	1	9/9/98	10/2/98	ND NJ	B

В

The MRL is elevated because of matrix interferences

Approved By

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# COLUMBIA ANALYTICAL SERVICES, INC.

# Analytical Report

Client:

Roy F. Weston, Inc

Project:

Duwamish River/4000-027-001-2019-38

Sample Matrix:

Sediment

Service Request: K9805975

Date Collected: 8/31/98

Date Received: 9/1/98

# Organochlorine Pesticides

Sample Name

98364012

Lab Code

K9805975-011

Units ug/Kg (ppb) Basis. Dry

Test Notes

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
alpha-BHC	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
beta-BHC	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
gamma-BHC (Lindane)	EPA 3550A	8081A	1	1	9/9/98	10/2/98	EN DN	
Heptachlor	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Aldrın	EPA 3550A	`8081A	1	1	9/9/98	10/2/98	ND	
Heptachlor Epoxide	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
gamma-Chlordane	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND UI	₽′
Endosulfan I	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	•
alpha-Chlordane	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Dieldrin	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND WI	
4,4'-DDE	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Endrin	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND WJ	
Endosulfan II	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
4,4'-DDD	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
Endrın Aldehyde	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
Endosulfan Sulfate	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
4,4'-DDT	EPA 3550A	8081A	2	1	9/9/98	10/2/98	4 J	
Endrin Ketone	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
Methoxychlor	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Toxaphene	EPA 3550A	8081A	60	1	9/9/98	10/2/98	ND UI	B

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The MRL is elevated because of matrix interferences

Approved By

1S22/020597p

Date 10/14/85

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